

**DETERMINATION OF ENDOTHALL IN DRINKING WATER BY ION EXCHANGE EXTRACTION, ACIDIC METHANOL METHYLATION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY**  
**EPA Method 548.1 Revision 1.0**

Facility Name: \_\_\_\_\_ VELAP ID: \_\_\_\_\_

Assessor Name: \_\_\_\_\_ Analyst Name: \_\_\_\_\_ Inspection Date: \_\_\_\_\_

| Relevant Aspect of Standards   | Method Reference | Y | N | N/A | Comments |
|--|------------------|---|---|-----|----------|
| <i>Records Examined:</i> SOP Number/ Revision/ Date _____ Analyst: _____<br>Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____   |                  |   |   |     |          |
| Does the laboratory have records of the<br>[ ] initial demonstration of capability and<br>[ ] continuing demonstrations of performance?  | 9.3              |   |   |     |          |
| Does the laboratory have records of the initial calibration of the instrument?   | 10.3             |   |   |     |          |
| Are stock standard solutions replaced after 6 months or sooner if comparison with check standards indicates a problem?   | 7.12.4           |   |   |     |          |
| Are records of the GC/MS tuning solution (5ng/μL DFTPP) analysis available?  | 10.3.2           |   |   |     |          |
| Is the injection volume 1 μL for each calibration standard?  | 10.3.4           |   |   |     |          |
| Is a response factor (RF) for endothall calculated for each calibration standard?  | 10.3.5           |   |   |     |          |
| Are the mean RF and the %RSD calculated for each analyte and surrogate?  | 10.3.5.1         |   |   |     |          |
| If the %RSD > 30%, is remedial action taken and the calibration repeated prior to analyzing samples?   | 10.3.5.1         |   |   |     |          |
| If the mean RF and %RSD were not calculated, was a regression curve generated from the calibration data?   | 10.3.6<br>12.1.2 |   |   |     |          |
| Check the file names and dates. Were all calibration standards analyzed on the same day? _____.<br>If no, why? _____   |                  |   |   |     |          |
| Are continuing calibration checks (CCV) performed at the beginning of each 8-hour work shift?  | 10.4             |   |   |     |          |
| Does the continuing calibration verification include a 1 μL injection of the GC/MS tuning solution?  | 10.4.1           |   |   |     |          |
| Does the continuing calibration verification include a 1 μL injection of mid-range calibration solution?   | 10.4.2           |   |   |     |          |
| Does the laboratory determine whether the area of the quantitation ion of the internal standard has decreased by more than 30% from the area measured at the most recent CCV, or by more than 50% from the area measured at initial calibration? | 10.4.3           |   |   |     |          |
| Notes  |                  |   |   |     |          |

Virginia Division of Consolidated Laboratory Services- Richmond, VA

| DETERMINATION OF ENDOTHALL IN DRINKING WATER BY ION EXCHANGE EXTRACTION, ACIDIC METHANOL METHYLATION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY<br>EPA Method 548.1 Revision 1.0  |                  |   |   |     |          |
|---|------------------|---|---|-----|----------|
| Relevant Aspect of Standards  | Method Reference | Y | N | N/A | Comments |
| If the area decreased by more than these amounts, were adjustments must be made to restore system sensitivity and the system recalibrated?  | 10.4.3           |   |   |     |          |
| Was the RF of endothall calculated and compared to the mean RF calculated in the initial calibration?   | 10.4.4           |   |   |     |          |
| Was the acceptance criteria $\pm 30\%$ of the mean RF calculated in the initial calibration?  | 10.4.4           |   |   |     |          |
| Are records maintained of the method blank analyzed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method?   | 9.2              |   |   |     |          |
| If within the retention time window of endothall, the reagent blank produces a peak which prevents the measurement of endothall, is the source of contamination determined and eliminated before processing samples?                                      | 9.2              |   |   |     |          |
| Does the analyst monitor the Internal Standard response of all samples during each analysis day to ensure that the IS response for any sample chromatogram does not deviate from the most recent calibration check standard IS response by more than 30%? | 9.5              |   |   |     |          |
| If the IS response deviates by more than 30%, does the analyst re-inject the sample?  | 9.5.1            |   |   |     |          |
| If the IS response of the re-injected sample is within 30% of the most recent calibration check standard IS response, is the result of the re-injected sample result reported?  | 9.5.1.1          |   |   |     |          |
| If the IS response of the re-injected sample deviates by more than 30%, and a sufficient volume of sample remains, does the analyst reprocess the sample?   | 9.5.1.2          |   |   |     |          |
| If consecutive samples fail the IS acceptance criterion, is a mid-range calibration check standard analyzed?  | 9.5.2            |   |   |     |          |
| If the RF of the check standard deviates from the predicted value by more than 20%, is the instrument recalibrated?   | 9.5.2.2          |   |   |     |          |
| Does the laboratory analyze at least one laboratory fortified blank (LFB) [or LCS] per sample set?  | 9.6.1            |   |   |     |          |
| If the LFB is judged to be out of control, is the cause identified and resolved before continuing analyses?   | 9.6.1            |   |   |     |          |
| Does the laboratory calculate control limits based on LFB recovery data and maintain control?   | 9.6.2            |   |   |     |          |
| Does the laboratory periodically determine and document its detection limit capabilities for endothall?   | 9.6.3            |   |   |     |          |
| Does the laboratory analyze a quality control sample from a source other than that used for the calibration standards at least once each quarter?   | 9.6.4            |   |   |     |          |

**DETERMINATION OF ENDOTHALL IN DRINKING WATER BY ION EXCHANGE EXTRACTION, ACIDIC METHANOL METHYLATION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY**  
**EPA Method 548.1 Revision 1.0**

| Relevant Aspect of Standards  | Method Reference    | Y | N | N/A | Comments |
|---|---------------------|---|---|-----|----------|
| Does the laboratory add a known fortified concentration [spike], at the same level used for the LFB, to a minimum of 10% of samples or one matrix sample per set, whichever is greater?   | 9.7.1               |   |   |     |          |
| Is the spike recovery compared to the control limits established for the LFB?   | 9.7.2               |   |   |     |          |
| If the spike recovery falls outside the designated range, but laboratory performance for that sample set is shown by the LFB recovery to be in control, the recovery problem with the fortified sample is judged to be matrix related, not system related. In such cases, is the result of the corresponding unfortified sample labeled to inform the data user that the results are suspect due to matrix effects? | 9.7.3               |   |   |     |          |
| <b>Anion Exchange Cartridge Preparation</b>   |                     |   |   |     |          |
| Are 8 mL anion exchange cartridges prepared using 50% v/v slurry of Bio-Rex 5 resin in reagent water?   | 11.1.1<br>11.1.2    |   |   |     |          |
| Is the final height of the wet resin bed in each cartridge 3.5±0.1 cm after excess water is drawn off under vacuum?   | 11.1.3              |   |   |     |          |
| Is a fritted disk placed horizontally atop the resin bed and pressed firmly into the bed to prevent sample from channeling around the disk?   | 11.1.4              |   |   |     |          |
| Are the prepared cartridges filled with water and half of the water drawn into the resin for storage until the cartridges are used?   | 11.1.4              |   |   |     |          |
| If the laboratory uses liquid-solid extraction disks, does the data meet the quality control criteria specified in section 9 of the reference method?   | Note at end of 11.1 |   |   |     |          |
| <b>Sample Preparation</b>   |                     |   |   |     |          |
| Are samples containing elevated levels of Ca <sup>2+</sup> , Mg <sup>2+</sup> or sulfate treated by adding 186 mg EDTA per 100 ml of sample to minimize interferences from those ions?  | 11.2.1              |   |   |     |          |
| Are samples diluted if interfering ions are present at levels that are not mitigated by the addition of EDTA?   | 11.2.2              |   |   |     |          |
| If laboratory practice is to dilute all samples, has the analyst demonstrated the ability to achieve an MDL of 2 µg/L or lower?   | 11.2.4              |   |   |     |          |
| Notes   |                     |   |   |     |          |

**DETERMINATION OF ENDOTHALL IN DRINKING WATER BY ION EXCHANGE EXTRACTION, ACIDIC METHANOL METHYLATION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY**  
**EPA Method 548.1 Revision 1.0**

| Relevant Aspect of Standards  | Method Reference | Y | N | N/A | Comments |
|---|------------------|---|---|-----|----------|
| <b>Sample Extraction</b>  |                  |   |   |     |          |
| Is the resin cartridge conditioned by drawing the following reagents through the cartridge in the following order?<br>1. 10 mL methanol<br>2. 10 mL reagent water<br>3. 10 mL 10% H <sub>2</sub> SO <sub>4</sub> in methanol<br>4. 10 mL reagent water<br>5. 20 mL 1 N NaOH<br>6. 20 mL reagent water | 11.3.2           |   |   |     |          |
| Is a 1 cm layer of each reagent retained in the cartridge to prevent the resin bed from drying out between steps?   | 11.3.2           |   |   |     |          |
| Is the sample reservoir filled with 60 mL of sample and the sample flow rate adjusted to 3 mL/minute?   | 11.3.3           |   |   |     |          |
| Is the balance of the sample added when needed to prevent the reservoir from going dry?   | 11.3.3           |   |   |     |          |
| After the sample passes through the cartridge, are the reservoir and adapter removed from the apparatus and a culture tube positioned inside the manifold to collect the eluent?  | 11.3.4           |   |   |     |          |
| Is 10 mL of methanol drawn through the resin cartridge making sure that any visible water inside the cartridge dissolves in the methanol?   | 11.3.4           |   |   |     |          |
| Is room air then drawn through the cartridge for five minutes under a vacuum of 10-20 in. Hg?   | 11.3.4           |   |   |     |          |
| Is the cartridge then eluted with 8 mL of 10% H <sub>2</sub> SO <sub>4</sub> in methanol followed by 6 mL of methylene chloride under vacuum over a one minute period?  | 11.3.5           |   |   |     |          |
| <b>Sample Derivatization and Partition</b>  |                  |   |   |     |          |
| Is the culture tube capped and held at 50°C for one hour in a heating block or water bath, then removed from the heat and allowed to cool for 10 minutes?   | 11.4.1           |   |   |     |          |
| Are the contents of the culture tube quantitatively transferred to a 125 mL separatory funnel by rinsing the tube with two x 0.5 mL aliquots of methylene chloride which are added to the separatory funnel?  | 11.4.2           |   |   |     |          |
| Is 20 mL of 10% sodium sulfate in reagent water added to the separatory funnel and the funnel shaken vigorously three times, venting with the stopcock?   | 11.4.2           |   |   |     |          |
| Is the funnel shaken for an additional 15 seconds and the phases then allowed to separate?  | 11.4.2           |   |   |     |          |
| Notes   |                  |   |   |     |          |

**DETERMINATION OF ENDOTHALL IN DRINKING WATER BY ION EXCHANGE EXTRACTION, ACIDIC METHANOL METHYLATION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY**  
**EPA Method 548.1 Revision 1.0**

| Relevant Aspect of Standards  | Method Reference | Y | N | N/A | Comments |
|---|------------------|---|---|-----|----------|
| After the phases have separated, is the lower organic layer drained into a 15 mL graduated centrifuge tube?   | 11.4.2           |   |   |     |          |
| Is the extraction repeated with two additional 2 mL aliquots of methylene chloride, adding the organic phase to the centrifuge tube each time?  | 11.4.2           |   |   |     |          |
| Is the extract fortified with 250 µL of the internal standard working solution (10 µg/mL Acenaphthene-d10) and concentrated to a final volume of 1.0 mL under dry nitrogen?           | 11.4.3           |   |   |     |          |
| <b>Chromatography</b>   |                  |   |   |     |          |
| Do the chromatography conditions conform to method specifications per EPA 548.1 Table 1 below?  | 11.4.4           |   |   |     |          |
| If other columns, detectors, or chromatographic conditions are used, has the laboratory demonstrated the ability to meet the requirements of the initial demonstration of capability? | 9.3<br>11.4.4    |   |   |     |          |
| Is endothall identified by comparison of its mass spectrum to a reference spectrum in a user created spectral library?  | 11.5.1           |   |   |     |          |
| Are FID identifications from the primary column verified by comparison to retention times on the confirmation column?   | 11.5.1           |   |   |     |          |
| If FID is used, do chromatographic conditions produce complete resolution of endothall?   | 12.1             |   |   |     |          |

**17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA**

**TABLE 1. RETENTION TIMES AND METHOD DETECTION LIMITS**

| Compound         | Column A | <u>Retention Time (min)</u> |          | <u>Method Detection Limit</u> |                   |     |
|------------------|----------|-----------------------------|----------|-------------------------------|-------------------|-----|
|                  |          | Column B                    | Column C | GC/MS                         | µg/L <sup>1</sup> | FID |
| Endothall        | 16.02    | 19.85                       | 18.32    | 1.79                          |                   | 0.7 |
| d10-Acenaphthene | 14.69    |                             |          |                               |                   |     |

<sup>1</sup>Based on seven replicate analyses of a reagent water fortified at 2 µg/L

Column A: [ ] DB-5 fused silica capillary for GC/MS, 30 m x 0.25 mm, 0.25 micron film  
 [ ] MS inlet temperature = 200°C  
 [ ] Injector temperature = 200°C  
 [ ] Temperature Program: Hold five minutes at 80°C, increase to 260°C at 10°/min, hold 10 minutes.

Column B: [ ] FID primary column, RTX Volatiles, 30 m x 0.53 mm I.D., 2 micron film thickness.  
 [ ] Detector temperature = 280°C  
 [ ] Injector Temperature = 200°C  
 [ ] Carrier gas velocity = 50 cm/sec.  
 [ ] Temperature program: Same as Column A

Column C: [ ] FID confirmation column, DB-5, 30 m x 0.32 mm ID, 0.25 micron film.  
 [ ] Carrier Gas velocity = 27 cm/sec  
 [ ] Same injector, detector and temperature program as Column A